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LIST OF PUBLICATIONS AND PATENTS WITH ABSTRACTS PERTAINING TO RESEARCH ON NATURAL RUBBER--1944-1949

Research on natural rubber in the Bureau of Agricultural and Industrial Chemistry has been conducted in three places in recent years. In the list below these places are indicated by letters as follows:

ERRL - Eastern Regional Research Laboratory
Chestnut Hill Station
Philadelphia 18, Pennsylvania

SRRL - Southern Regional Research Laboratory
2100 Robert E. Lee Boulevard
New Orleans 19, Louisiana

NREPI- Natural Rubber Extraction & Processing
Investigations, 1636 East Alisal Street,
Salinas, California. Formerly Guayule
Rubber Extraction Research Unit.

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Bureau of Agricultural and Industrial Chemistry
Agricultural Research Administration
UNITED STATES DEPARTMENT OF AGRICULTURE

LIST OF PUBLICATIONS AND PATENTS, WITH ABSTRACTS, PERTAINING TO RESEARCH ON NATURAL RUBBER

1944

DECOMPOSITION OF GUAYULE RESINS BY MICROORGANISMS. P. J. Allen, J. Naghski, and S. R. Hoover (ERRL). Jour. Bact. 47(6):559-572. A method is described for detecting resin-decomposing organisms by their ability to cause clearing of resin emulsions suspended in agar. A number of resin-decomposing organisms were isolated. The rate and extent of clearing of resin-agar emulsions are correlated with decomposition of resin in liquid culture, as shown by chemical analysis. Certain constituents are decomposed selectively. Presence of nutrients does not prevent production of resin-decomposing enzymes, but on the contrary, by increasing growth, leads to more rapid and extensive clearing.

AEROBIC DECOMPOSITION OF GUAYULE SHRUB (PARTHENIUM ARGENTATUM GRAY). J. Naghski J. W. White, Jr., and S. R. Hoover (ERRL). Jour. Bact. 48(2):159-178. Aerobic decomposition of crushed guayule shrub was carried out at 36, 44, and 53°C. for one to three weeks. A 20 to 30 percent loss of dry weight, with marked softening and disintegration of woody tissue, was observed. The profuse microflora was tested for its biochemical reactions, and the predominating organisms were identified. The disappearance of water-soluble carbohydrates and existence of a saccharalytic flora were correlated with the markedly thermogenic phase during the first week. A mixed neutral and proteolytic flora followed during the second and third weeks.

ISOLATION OF PARTHENIOL, PARTHENYL CINNAMATE, AND OTHER CONSTITUENTS FROM GUAYULE RESIN. E. D. Walter (ERRL). Jour. Amer. Chem. Soc. 66(3):419-421. Partheniol, a new sesquiterpene alcohol, and parthenyl cinnamate were isolated from guayule resin, and some of their properties, including crystallographic optical properties, are described. A crystalline waxlike substance and several fractions of volatile oils, probably sesquiterpenes, were prepared from acetone extracts of whole shrub, guayule foliage, and guayule rubber.

OIL BLUE NA AS A STAIN FOR RUBBER IN SECTIONED OR GROUND PLANT TISSUES. R. T. Whittenberger (ERRL). Stain Technol. 19(3):93-98. Oil blue NA (Calco), a stain which colors rubber bright blue, was used in studying the distribution of rubber in several plant species. Fresh or fixed sections are cut, bleached with Javelle water or NaOCl solution, treated with 9 percent KOH in 95 percent ethanol, washed with several changes of water, and finally with 95 percent ethanol, and stained with 0.05 percent oil blue NA in 70 percent ethanol. Sections are rinsed in 50 percent ethanol, placed in 40 percent glycerin, and mounted in glycerin jelly. A microscopic technique is presented whereby it is possible to determine approximately the relative proportion of dispersed and coagulated rubber in unstained tissues.

EXTRACTION, CHARACTERIZATION, AND UTILIZATION OF GOLDENROD RUBBER (SRRL) Parts I-II. Processed (no date). The complete experimental data including tables, graphs, drawings, pictures, and discussions regarding investigations conducted on goldenrod rubber at the Southern Regional Research Laboratory from July 1942 to June 1944 are presented in 17 sections. Typical subjects covered are: preparation or treatment of raw material by retting, chemical methods, ensilage, antioxidant, drying, grinding, and separation and screening; laboratory and pilot-plant development of extraction methods; methods of rubber purification; vulcanization, testing and processing; industrial processing and fabrication tests; analytical and physical-chemical investigations.

RETTING AS AN AID TO RUBBER EXTRACTION FROM GOLDENROD. J. J. Spadaro, E. F. Pollard, N. Porges (SRRL). Rubber Age (N.Y.) 56:53. The development of a procedure for anaerobic-aerobic retting of goldenrod plant material with diluted, digested sewage sludge is given. Retting aids in recovery of rubber from goldenrod by reducing dry weight of material 21 to 40 percent and resin content by about 50 percent. Extraction time was decreased by 1/4 to 1/3 of that previously required. Rubber quality was superior to that obtained from unretted material.

A SOLUTION METHOD FOR THE COMPOUNDING OF GOLDENROD RUBBER. F. L. McKennon and J. R. Linquist (SRRL). Rubber Age (N.Y.) 56:289-292. Republished Rubber Chem. and Tech. 18:679-84 (1945). A solution method of compounding rubber has been devised, particularly suitable for compounding a soft natural rubber such as that recovered from goldenrod, but it does not appear to be restricted to goldenrod rubber. No particular inherent disadvantage of solution compounding is indicated with Hevea rubber. By proper choice of solvent and compounding chemicals, the possibility is presented of improving quality of those rubbers in which proper dispersion of chemicals is difficult. Also shows possibilities of being a valuable research tool in the pretreatment of soft synthetic elastomers as a preliminary step to evaluation of rubber-like qualities.

ISOLATION OF QUERCITRIN AND QUERCETIN FROM GOLDENROD MATERIAL. J. C. Guthrie, M. F. O'Connor, M. F. Stansbury, T. R. Savich (SRRL). Jour. Amer. Chem. Soc. 66:1794. Quercitrin and quercetin were isolated from the acetone extractives of goldenrod material. The quercetin in 95 percent ethanol showed absorption maxima at 2570 and 3750 Å. with extinction coefficients of 67.0 and 75.5, respectively.

1945

THE COMPOUNDING OF GUAYULE RUBBERS--EFFECT OF CARBON BLACK LOADINGS. F. E. Clark and W. F. L. Place (NREPI). India Rubber World 112:67-72. Hevea smoked sheet was superior to any of the guayule rubbers tested from an overall consideration of physical properties. Latex guayule and deresinated guayule, however, represent major improvement over the ordinary resinous guayule. Retted guayule shows some improvement over resinous rubbers, particularly with respect to tensile strength, although the improvement is not so noticeable in other properties, such as abrasion resistance.

CRYPTOSTEGIA LEAF RUBBER. S. R. Hoover, T. J. Dietz, J. Naghski, and J. W. White, Jr. (ERRL). Indus. and Engin. Chem. 37(9):803-809. The leaves of *Cryptostegia grandiflora* were investigated as a possible emergency source of rubber, supplementing the latex rubber from the stems. The leaf rubber consists of two distinct portions; 85 to 90 percent is a relatively low-polymer rubber which occurs in the chlorenchyma cells, and 10 to 15 percent is a latex rubber of better quality which occurs in the latex ducts. Isolation of the leaf rubber by a combination of fermentation and chemical extraction and the characterization of vulcanizates are described.

ISOLATION OF BETAINE FROM GUAYULE. C. W. Murray and E. D. Walter (NREPI). Jour. Amer. Chem. Soc. 67:1422. Betaine was isolated from the water-soluble fraction of the leaves from defoliated guayule shrub and from crude pebble-milled rubber. Betaine was also obtained from the product of each step in the extraction of the rubber.

ANAEROBIC FERMENTATION OF CRYPTOSTEGIA LEAVES FOR RECOVERY OF RUBBER. J. Naghski J. W. White, Jr., S. R. Hoover, and J. J. Willaman (ERRL). Jour. Bact. 49(6):563-574. A two-day anaerobic decomposition of Cryptostegia leaves by *Clostridium roseum* at 35 to 45°C. effected a loss of 60 percent in the original dry weight of leaves and a 75 percent loss in the crude cellulose. The rubber content of the product was more than two and one-half times that of the original material, on a moisture-free basis.

MICROSCOPICAL STUDIES IN CONNECTION WITH THE EXTRACTION OF RUBBER FROM GOLDENROD. M. L. Rollins, T. L. W. Bailey, Jr., and L. V. DeGruy (SRRL). India Rubber World 113:75-78. Microscopical studies, aided by histological staining techniques, were made to demonstrate the location of rubber in the chlorophyllous cells of the goldenrod leaf and the relative non-occurrence of rubber in all other parts of the plant. Extension of these techniques to control studies of laboratory and pilot-plant procedures made it possible to follow microscopically the various steps in the development of a method for the extraction of rubber. Microscopical examinations of raw materials and processed residues showed that the rubber in the leaf is completely removed by an acetone-benzene extraction process if the raw material is finely ground before extraction.

PHYSICAL-CHEMICAL INVESTIGATIONS OF GOLDENROD RUBBER. IV. INCREASE IN VISCOSITY AND FORMATION OF PHOTOGELS BY IRRADIATION OF GOLDENROD RUBBER IN THE ABSENCE OF OXYGEN. M. M. Graff and E. L. Skau (SRRL). Jour. Phys. Chem. 49(1):1-4. Republished in Rubber Chem. and Tech. 18:685-87. The viscosities of irradiated benzene solutions are determined in the presence of various activators. Gelling times in sunlight, ranging from less than 4.5 hours for benzophenone to 108 hours for quinone, together with precipitation values, are given. Benzophenone, benzaldehyde, and carbon tetrachloride seem to be the best of the activators tested.

PHYSICAL-CHEMICAL INVESTIGATIONS OF GOLDENROD RUBBER. I. "PRECIPITATION VALUE" AND ITS SIGNIFICANCE IN CHARACTERIZATION AND EVALUATION OF NATURAL RUBBERS. E. L. Skau, W. J. Runckel, F. B. Kreeger, and M. A. Sullivan (SRRL). Jour. Phys. Chem. 49(4):281-94. A simple titration method was developed for determining the "precipitation value" of a sample of rubber. Precipitation values and viscosities were determined on goldenrod rubber from various pilot-plant runs, on a number of different kinds of rubber, and on various binary mixtures of these and other samples of rubber partially degraded by irradiation. It is indicated that the precipitation value--and therefore the solubility--of a rubber specimen is determined by the fraction of highest molecular weight that is present in appreciable quantity.

PHYSICAL-CHEMICAL INVESTIGATIONS OF GOLDENROD RUBBER. II. THE PRECIPITATION VALUE AS AFFECTED BY IRRADIATION IN THE PRESENCE OF VARIOUS RUBBER ANTIOXIDANTS AND OTHER REAGENTS. E. L. Skau, F. B. Kreeger, and W. J. Runckel (SRRL). Jour. Phys. Chem. 49(4):295-303. The precipitation value was used to investigate the light degradation of goldenrod rubber in benzene solution as affected by the presence of rubber antioxidants and other reagents. Of the antioxidants tested, p-aminophenol was the most effective in inhibiting the light reaction. Phenyl-beta-naphthylamine behaved consistently as an activator. It is apparent that the inhibiting effect of antioxidants as estimated by this method is not a measure of the effectiveness of rubber antioxidants in the ordinary sense.

PHYSICAL-CHEMICAL INVESTIGATIONS OF GOLDENROD RUBBER. III. THE FRACTIONATION OF GOLDENROD AND OTHER NATURAL RUBBERS. E. L. Skau, W. J. Runckel, F. B. Kreeger, and M. A. Sullivan (SRRL). Jour. Phys. Chem. 49(4):304-315. A method for fractionation of rubber by successive precipitations from a benzene solution was investigated and applied to various samples of goldenrod and other varieties of natural rubbers. The size of each successive fraction and its intrinsic viscosity were determined. Histograms were drawn which characterized the molecular-weight distribution of the polymer. The area of the histogram should be equal to the average molecular weight and generally checked reasonably well with the value as determined experimentally. The percentage of Hevea-goldenrod rubber mixtures can be estimated from histogram areas.

IMPROVING GUAYULE RUBBER BY SHRUB RETTING. J. W. White, Jr., J. Naghski, F. J. Allen, and S. R. Hoover (ERRL). India Rubber World 111(5):570-573. Forced aeration made it possible to ret guayule shrub under packing conditions equivalent to a depth of at least 5.2 feet. This aerobic retting improved the properties of the rubber, in confirmation and extension of the results obtained by Spence, who did not use forced aeration. Under the conditions employed (adequate aeration, 60 to 70 percent moisture, and 104° to 129°F.) about 1200 p.s.i. improvement in tensile strength of guayule rubber was obtained by retting the shrub for 21 days, with proportionately less increase in shorter time. The resin content of the recovered rubber was reduced from 20-23 to 12.3-13.5 percent by the process.

URSOLIC ACID AND PARAFFIN HYDROCARBONS FROM CRYPTOSTEGIA LEAVES. J. W. White, Jr. and F. R. Senti (ERRL). Jour. Amer. Chem. Soc. 67(5):881-82. Ursolic acid was isolated from a hot acetone extract of leaves of Cryptostegia grandiflora which had been given a fermentation pretreatment for rubber recovery. Three fractions consisting of different paraffin hydrocarbons were obtained from a neutral portion of the acetone extract after saponification.

DISTRIBUTION OF RUBBER IN CRYPTOSTEGIA AS A FACTOR IN ITS RECOVERY. R. T. Whittenberger, B. A. Brice, and M. J. Copley (ERRL). India Rubber World 112(3): 319-23. Rubber occurs in Cryptostegia both in a laticiferous duct system and in individual leaf chlorenchyma cells. The ducts occur principally in the stems, but extend into the leaves, paralleling the vein system. Dissection and chemical analyses indicate that 85 to 90 percent of the rubber in a mature Cryptostegia hybrid leaf exists in the chlorenchyma cells and the remaining 10 to 15 percent in the ducts.

RUBBER IN CRYPTOSTEGIA LEAF CHLORENCHYMA. R. T. Whittenberger and A. Kelner (ERRL). Amer. Jour. Bot. 32(10):619-627. In mature Cryptostegia hybrid leaves, 85 to 90 percent of the total rubber is in the chlorenchyma, the remaining 10 to 15 percent occurring as latex in the laticiferous ducts. Proof that the leaf chlorenchyma globules contained rubber was established by X-ray studies of the isolated globules. The chlorenchyma rubber is apparently of lower molecular weight than that of the laticiferous ducts. The globules contain about 65 percent rubber hydrocarbon, the remaining 35 percent consisting largely of acetone-soluble material (resins).

1946

EVALUATION OF BLENDS OF GUAYULE AND GR-S RUBBERS. F. E. Clark and W.F.L. Place (NREPI). Indus. and Engin. Chem. 38:1026. Advantages derived from blending small quantities of guayule rubbers with the all-purpose synthetic rubber, GR-S, are demonstrated. The lower-resin guayule rubbers, used in amounts up to 20 percent of the total rubber, produce more improvement in the physical properties of the blends than does the guayule of higher resin content. The latter, however, produces a greater improvement in processing characteristics than does the former.

A NOVEL HIGH PRESSURE TREATMENT. J. M. Cumming and R. L. Chubb (NREPI). Chem. and Met. Engin. 53:125-126. Equipment for rapid and semi-continuous waterlogging of cork to facilitate removal of cork particles from rubber in connection with the conventional flotation process following pebble milling of guayule is described, Hydraulic pressure of 2,000 pounds per square inch is applied for 20 seconds to effect waterlogging.

NATURAL RUBBER FROM RUSSIAN DANDELION. R.K. Eskew (ERRL). India Rubber World 113:517-520. A pilot-plant-scale process for recovering rubber from kok-saghyz (Russian dandelion) roots is described. The roots are leached to remove carbohydrates, pebble-milled with water to crush them and agglomerate the rubber, and screened to remove the fleshy part of the roots from the rubber and root skins. The latter are again pebble-milled with water to completely disengage the rubber from the skins; a flotation step then separates the rubber from the skins. The rubber obtained is of high quality as shown by performance tests in tires constructed of kok-saghyz rubber by commercial rubber companies.

PROCESS FOR RECOVERING RUBBER FROM FLESHY PLANTS. Patent 2,393,035 to R. K. Eskew and Paul W. Edwards. Patented Jan. 15, 1946. Russian dandelion roots are leached to remove carbohydrates, then milled with water to crush them and agglomerate the rubber, then screened to separate the fleshy part of the roots from the rubber and root skins. The rubber and skins are again milled with water to disengage completely the rubber from the skins. Then a flotation step separates the rubber and the skins.

TREATMENT OF RUBBER. Patent 2,399,156 to Paul Stenberger, R. K. Eskew, and Roy S. Hanslick. Patented April 23, 1946. A raw resinous rubber such as guayule is treated with a hot mettalic hydroxide solution, after which it is scrubbed with a sufficient quantity of a hot water dispersion of a higher fatty acid to neutralize the remaining alkali. The rubber is then washed and dried. An extraction with ethyl alcohol may precede the hot alkali treatment.

GUAYULE RUBBER BY FERMENTATION. Patent 2,408,853 to S. R. Hoover, Paul J. Allen, and Joseph Naghski (ERRL). Patented Oct. 8, 1946. Fermentation of guayule rubber worms in aqueous medium with *Pseudomonas boreopolis*, *Aspergillus fumigatus*, or *Trichoderma sp.* of microorganisms results in an improved guayule rubber characterized by lower resins and higher tensile strength.

THE EVALUATION OF GUAYULE RUBBER--DEVELOPMENT OF TESTING FORMULAE. W.F.L. Place and F. E. Clark (NREPI). India Rubber World 115:370-73. Noticeable improvement of guayule rubber can be effected through correct compounding. Resinous guayule requires more sulfur and stearic acid than the ordinary Hevea rubber. The non-rubber constituents of resinous guayule rubber react with compounding ingredients during vulcanization. Standard vulcanization formulas are therefore not suitable for guayule rubber. Further study of guayule rubber-compounding requirements to obtain best physical properties of vulcanizates is recommended.

METHODS OF APPLYING CERTAIN ANTIOXIDANTS TO GUAYULE CRUDE RUBBER FOR PRESERVATION DURING STORAGE. H. Tint and J. M. Cunning (NREPI). India Rubber World 113: 509-512, 524. It was shown that the primary aromatic amine, tonox, can be applied to wet rubber at a concentration of 0.5 percent (dry-rubber basis) colloidally dispersed in water and applied as a spray, to yield the same stiffening effect on the product heretofore obtained by spraying the same concentration as an acetone solution. The newer method is less hazardous and more economical.

RAPID ESTIMATION OF RUBBER IN GUAYULE LATEX DISPERSIONS. R. T. Whittenberger and B. A. Brice (ERRL). Indus. and Engin. Chem., Analyt. Ed. 18:209-210. A microscopic method for estimating rubber content of latex dispersions, based essentially upon the identification and counting of latex particles in a small volume of dispersion, is described. With appropriate standardization this method may serve a variety of purposes for which chemical methods of analysis would be too time-consuming.